

Process Optimization of BCl_3/Cl_2 Plasma Etching of Aluminum with Design of Experiment (DOE)

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Abstract

Reactive ion etching of aluminum (Al) had been carried out with BCl_3/Cl_2 , and process optimization was performed using statistical process modeling technique. I-optimal design was employed to set up the etch experiment with operating parameters, namely, gas composition, RF power, and chamber pressure. Analysis has been performed on etch rate, selectivity, and profile, individually, and the process optimization including three responses of interest, simultaneously. Through the experiment we have confirmed that etch rate was affected by Cl_2 , but the excessive amount of chlorine decreased etch rate. BCl_3 is significantly related with selectivity, and increased power gives good selectivity because RF power range was from 130 to 170 watts. Selectivity was affected by how to fast remove Al_2O_3 by BCl_3 . Etch profile was strongly related with all the three parameters, and they affect on anisotropic sidewall property. Suggested optimization simultaneously considers three responses of interests, which is crucial in process development and optimization for quickly ramping up high volume manufacturing.

Introduction

The present semiconductor fabrication equipments used at a university laboratory are superannuated or utilized relatively for a long period due to its high cost. Although the equipments have a good performance at the beginning of purchase, processing performance is gradually degraded as years over. When experimenters are carried out experiment with existing recipe, obtaining desired result of an experiment is sometimes difficult. In such a case the following question can be arisen; what was the method that reduces an error and predicts a range of fluctuation of the result. In this paper, we will focus our attention on aluminum (Al) etch, which is the most relevant material in interconnects in semiconductor fabrication. Several metallic materials such as aluminum and its alloys, tungsten, copper, platinum are frequently utilized in not only the interconnection layer but also the device packaging process of the semiconductor manufacturing industry. Choice of material is important for achieving a desired interconnection layer condition for better transmission of electrical signal. Material is selected based on its essential properties, such as electrical conductivity, mechanical and chemical stability, adhesion characteristics, film deposition considerations, and patternability [1]. Although copper is increasingly used for metallization process in current leading edge semiconductor manufacturing, aluminum has been generally used for an interconnection material in integrated circuits (IC) because of its bondability to Si and SiO_2 and easiness for patternability than other metals. The fluorine containing gases used to etch Si and SiO_2 are not suitable for etching Al since etch product, AlF_3 , has very low vapor pressure at temperatures below 100°C . AlCl_3 have sufficiently high vapor pressures to allow plasma etching of Al, and thus chlorinated gases have been exploited to develop dry etch process for aluminum thin film [2]. The most common gases are BCl_3/Cl_2 composition at Al etch. Chlorine is the primary etchant and BCl_3 act as a source of heavy ions for bombardment. Therefore, as the first step in our goal, we have studied how process optimization of mixed BCl_3/Cl_2 plasma etching of Al with design of experiment (DOE) in superannuated Oxford 80 plus. Process is optimized using a statistically established process model. A designed experiment was the simultaneous evaluation of two or more factors (parameters) for their ability to affect the resultant average or variability of particular product or process characteristics. I-optimal design was employed to set up the etching experiment with operating parameters. The second step we have confirmed that a model established through the analysis of DOE process analysis as far as it goes to predict the range of fluctuation of results of etch rate, profile, and selectivity.

Experiment

Al sputtered samples were prepared on a 4" silicon wafer without an inter-dielectric layer. The structure of samples used for these experiments is as follows: The Al layer was deposited at 6000 Å by a direct current (DC) magnetron sputtering at a pressure of 6 mTorr and a sputtering power of 150 watts. Patterned AZ1512 photoresist was used for the etch mask. **Figure 1** shows the structure of samples.

The experiment design suggests a number of experimental design schemes, including a central composite design and a Box-Behnken design for process optimization. In this paper, we have employed the I-optimal design to find an optimum operating condition or ranges of factors for which the response falls within an acceptable range. The focus is clearly on precise estimation of the predicted response, as opposed to precise estimation of the model parameters. By minimizing the average variance of prediction, I-optimal designs provide more precise estimates of the predicted response. This, in turn, leads to better location of the best response or an acceptable operating range [3]. The controllable factors consist of the amount of BCl₃ and Cl₂ gases, pressure, and RF power in the experiments.

According to the designed experiment, one set of 16 experiments with 2 augmented center points were performed while all three tool parameters were simultaneously varied. After processing, the three responses of interest, such as etch rate, selectivity and profile were measured by both μ -step and SEM. Then statistical analysis was quantitatively implemented, i.e. main and interaction effects, analysis of variance, and regression analysis.

Result and Discussion

Fitting a regression model of our optimization experiment requires several underlying assumptions. Estimation of the model parameters requires the assumption that the errors are uncorrelated random variables with mean zero and constant variance. The results of the Shapiro-Wilk test for normality which is reported when the sample size is less than 2000 are illustrated in Figures 2 and 3. The p-values of etch rate and selectivity were 0.097 and 0.142, respectively. The null hypothesis (H₀) is the data from the normal distribution. Small p-values rejected the H₀; however, the etch p-values were larger than 0.050, so data set was regarded as in normal distribution. The profile is not a normal distribution because p-value is 0.0026. So, the profile does not statistic analysis. After the normality test, we investigated the variables screening technique based on the stepwise regression, which selects suitable variables for regression by generating an adjusted R-squared (R²-adj) that indicates model accuracy.

There are two other ways to assess the overall adequacy of the model; R² and R²-adj. In this model, the values of R² and R²-adj are 0.958751 and 0.912346, respectively. The coefficient of multiple determinations, R² may be used as a global statistic to assess the fit of the model. The model accounts for about 95% of the variability in the efficiency response. The R² statistic is somewhat problematic as a measure of the quality of the fit for a multiple regression model because it always increases when a variable is added to a model. Since R² always increases when a predictor variable is added, it can be difficult to judge whether the increase is telling us anything useful about the new predictor variable. That is the reason why *R²-adj* is more popular for evaluating the fit of the model [4]. The value of R² and R²-adj was somewhat low, indicating that above prediction equation does not provide a perfect fit to real data. One of the future works would be to implement uncontrollable factors such as substrate temperature in the etch chamber.

The analysis of variance and the lack of fit test are presented in Table 2 and Table 3, respectively. The analysis of variance is the appropriate procedure for testing equality of several means is [5]. The lack of fit test is an implication to add more effects to the model using higher orders of the terms already in model. The lack of fit test is important for examining whether anything was left out of the model [6].

Regression analysis is a statistical technique for investigating and modeling the relationship between variables. An important objective of regression analysis is to build a mathematical model that will help make accurate predictions about the impact of variable variations [7]. This process is also called fitting the model to the data. In general, the response variable y may be related to k predictor variables, x_1, x_2, \dots, x_k . This is called a multiple linear regression model because more than one predictor variable is involved.

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \dots + \beta_k x_k + \varepsilon \quad (1)$$

To make a regression model, a term was added to represent the interaction between Cl₂ and RF power, square term of Cl₂, square term of pressure in the etch rate model due to a significant action in the model. The selectivity model involved interactions such as the BCl₃ and RF power in Equation (1).

Figure 4 shows that the contour plot based on etch rate. The selectivity regression model finds the ranges of the factors for which the response falls within the optimum operating condition in order to maximize both etch rate and profile. Based on the contour plot, we found that both dotted lines (etch rate) and solid lines (selectivity) are maximum regions. Summarizing, the ranges of the factors for the optimum condition are; Cl₂ gas from 8 to 10 sccm, BCl₃ gas from 40 to 45 sccm, pressure from 48 to 52 sccm, and RF power from 165 to 170W. In Figure 4, cross (×) points are mid-points of the optimum operating condition. The regression model estimated 2050 Å/min of etch rate and 7.2:1 degrees of selectivity at this operating condition.

Through the experiment, we confirmed that Al film reacting with chlorine makes volatile etch by-products such as aluminum trichloride (AlCl₃), which can determine etch rate: $2Al + 3Cl_2 \rightarrow AlCl_3$. To achieve an etching action with a chemical mechanism, BCl₃ provides bombarding positive ions that are accelerated toward Al₂O₃ on the Au film surface by strong electric fields. The ions of BCl₃ physically assist the protected Al film surface by a sputter-etch action. A benefit of this mechanical etching approach is the high etch rate, making it possible to achieve highly selectivity. That is, a combined physical and chemical mechanism where ions of BCl₃ bombardment improves the chemical etching action. So, BCl₃ is significantly related with selectivity, and increased power gives good selectivity because RF power range was from 130W to 170W. Selectivity was affected by how to fast remove Al₂O₃ by BCl₃. The etch profile is varied from isotropic to anisotropic by adjusting plasma conditions. Therefore, the profile is strongly related with two parameters such as BCl₃ and Cl₂, an increase of the its affect on anisotropic sidewall characteristics. Controlling the amount of carbon chloride yields a good etch rate with a satisfactory sidewall profile. Figure 5 shows the experiment performed to verify the regression model based on the mid-point of the optimum condition. Also by the medium of an analysis of statistical method by repeatedly carry out the optimum condition experiments, we predicted a range of fluctuation of etch rate 1930 ± 220 Å/min and selectivity 7.2 ± 2.4 , profile $75.1 \pm 10.4^\circ$, respectively.

As a result, we were able to achieve satisfactory results in Al film etch condition in terms of etch rate and selectivity. I confirmed that reduce and predict a range of fluctuation of etch rate and selectivity, profile, respectively.

Conclusion

In this paper, reactive ion etching of Al was performed with BCl₃/Cl gases, and process optimization was performed using a statistically established process model. As a result, we obtained a satisfactory Au film etch condition in terms of etch rate and selectivity, profile. Although the suggested optimization approach may not guarantee the best etch rate and sidewall profile, it is more suitable than the metal etch process. The optimized condition of the suggested statistical method can be applied to plasma etching of other noble metals, such as platinum and iridium, for an improved etching process. Also by the medium of statistical method, I confirmed that reduce and predict a range of fluctuation of etch rate and selectivity, profile, respectively.

References

01. P. Werbaneth, Z. Hasan, Pa. Rajora, and M. Rousey-Seidel. (1999), The International Conference on Compound Semiconductor Manufacturing Technology (1999) 1563.
02. S. Wolf, R.N Tauber Silicon processing; 2nd Ed.
03. J.P. Sall, SUGI 27, Orlando, Florida, U.S.A., April 14-17, 2002, SUGI Proceedings (2002) 268.
04. Phillip J. Ross, Taguchi Techniques for Quality Engineering; 2nd Ed. McGraw-Hill, 1996.
05. Douglas C. Montgomery, Design and Analysis of Experiments, Wiley, 2004.

06. John Sall, Lee Creighton, Ann Lehman, JMP Start Statistics, SAS, 2007.

07. I. Bass, Six Sigma Statistics with EXCEL and MINITAB, McGraw-Hill, 2007

PR etch mask



Figure 1. Sample structure

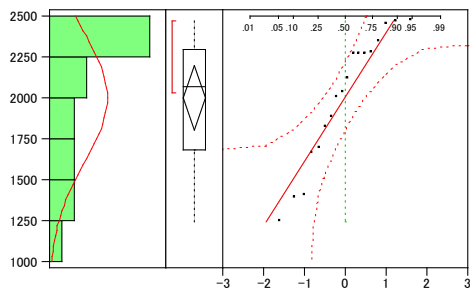


Figure 2. Quantile plot of etch rate

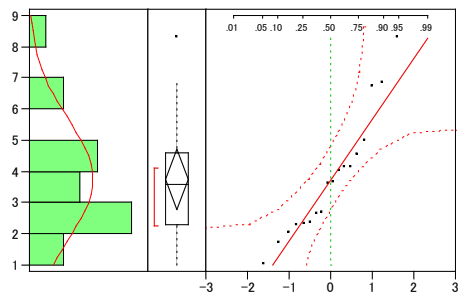


Figure 3. Quantile plot of sidewall profile

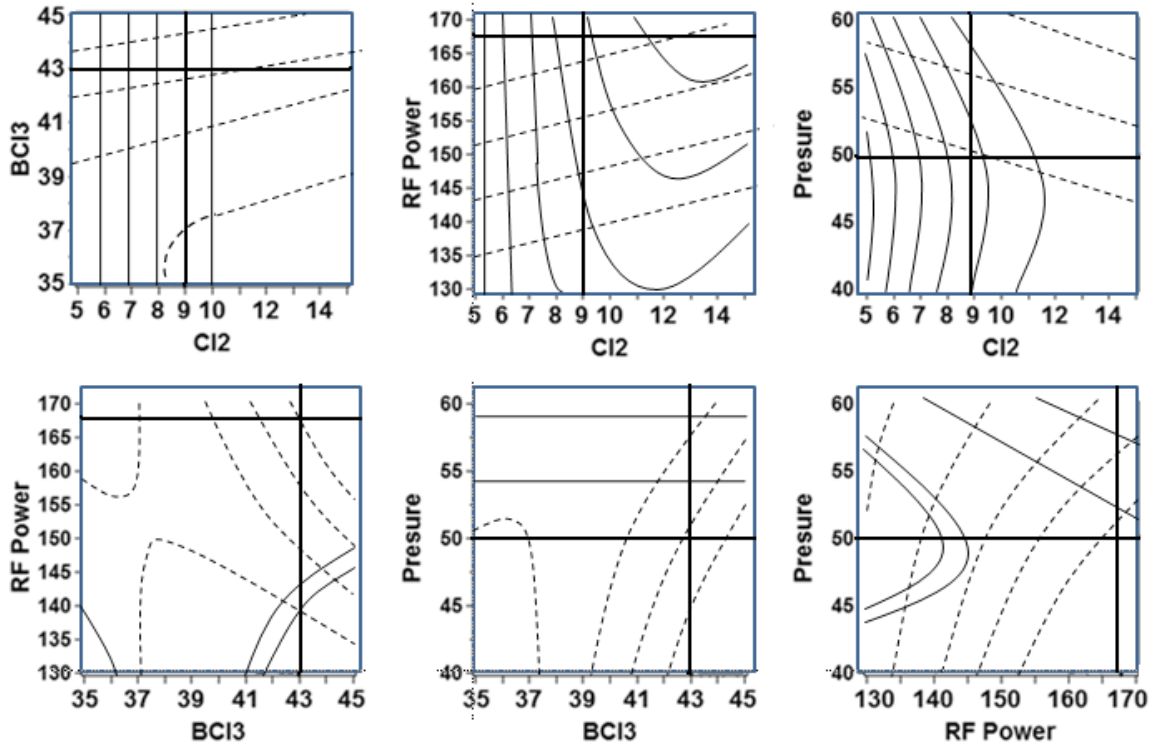


Figure 5. Verification experiment result of Au film etching

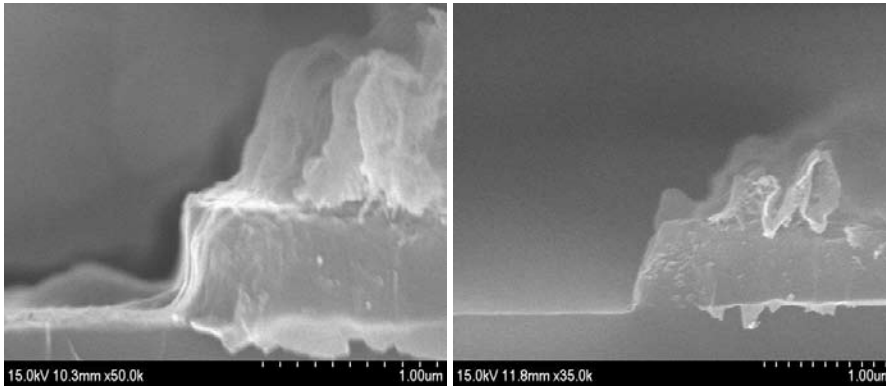


Table 1. Process parameters and ranges for the I-optimal design test

Parameters	Ranges		Unit
	Low	High	
BCI3	35	45	Scm
CI	5	15	Scm
Pressure	40	60	mTorr
RF Power	130	170	W

Table 3. Lack of fit test for etch rate

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	6	30767.24	5127.9	0.1321
Pure Error	2	77664.89	38832.4	Prob. > F
Total Error	8	108432.12		0.9772
				Max R Sq
				0.9705

Table 4. ANOVA table for selectivity

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	8	45.261563	5.6577	5.4311
Error	9	9.375500	1.04172	Prob. > F
C. Total	17	54.637063		0..0102*

Table 5. Lack of fit test for selectivity

Source	DF	Sum of Squares	Mean Square	F Ratio
Lack Of Fit	5	9.249060	1.84981	0.8133
Pure Error	9	20.471303	2.27459	Prob. > F
Total Error	14	29.72364		0.1175
				Max R Sq
				0.9940